



**PATENT**  
**72162-243315**

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of:	)	Art Unit: 1723
	)	Examiner: MENON, KRISHNAN
AGARWAL, ASHOK	)	
	)	
Serial No: 09/974,637	)	
	)	
Filed: October 9, 2001	)	
	)	
For: COMPOSITE NANOFILTRATION AND	)	
REVERSE OSMOSIS MEMBRANES AND	)	
METHOD FOR PRODUCING THE SAME	)	

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**DECLARATION OF JOHN SIMONETTI RE: UNEXPECTED RESULTS UNDER  
37 C.F.R. 1.132**

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Sir:

**I, John Simonetti, do hereby declare and state as follows:**

I. I graduated from San Diego State University in 1974, with a degree in Microbiology. I have been an employee of the assignee of the above-referenced patent application since 1993. I have been working in the field of filtration membranes since 1977. I am familiar with the prosecution history of the above-referenced patent application, including the Office Action dated February 17, 2004.

**II. Experiments:**

Under my direction the following experiments were conducted to determine the fluid fluxes of the inventive filtration membranes. These experiments clearly demonstrate that the fluid fluxes of the inventive filtration membranes are significantly higher compared to the fluid fluxes of the known membranes.

#### **Example 1: Preparation of Filtration Membrane**

A filtration membrane was prepared in accordance with the disclosure of the above-referenced application, using the following method:

- A. AS-100 Ultrafilter from PTI ADVANCED FILTRATION was used as the microporous substrate.
- B. An aqueous amine solution was prepared in the following manner.
1. Weigh 650.0g +/- 1.0g of sodium propionate and dissolve in 5.0L of deionized water (DIW).
  2. Weigh 450g +/- 1.0g piperazine flakes and add and dissolve the piperazine flakes into the sodium propionate solution.
  3. Pour salt/piperazine concentrate solution into 24.0L of DIW to make the aqueous phase coating solution.

#### **INFORMATION REGARDING CHEMICALS USED:**

Piperazine (1,4-Diethylenediamine Anhydrous), CAS 110-85-0, Spectrum, Product # Pi123.

Sodium Propionate (Propionic Acid Sodium Salt), CAS 137-40-6, Spectrum, Product # So192.

C. The microporous substrate was wetted with the aqueous amine solution by guiding the continuous membrane web into a bath containing the amine/salt solution so one side of the membrane contacts the liquid with the opposite side of the membrane web contacting a roller partially submerged in the liquid. The aqueous amine coated web is next exposed to an air knife or squeegee to remove excess liquid and subsequently guided to a solution of the organic phase.

D. The wetted substrate was brought into contact with an acyl halide solution comprising 33.0 grams of 1,3,5-Benzenetricarbonyl trichloride (trimesoyl chloride) in 30L of naphtha. Here again, only the amine-coated surface of the membrane web is exposed to the organic phase by guiding the web over a partially submerged roller.

#### **INFORMATION REGARDING THE CHEMICALS USED:**

1,3,5-Benzenetricarbonyl trichloride 98% (trimesoyl chloride), Aldrich, Cat # 14,753-2, CAS # 4422-95-1

Naphtha VM&P Rule 66, Vopak USA Inc., Product # 632631, CAS # 64742-89-8, 349 LB Drum.

E. Following contact with the organic phase, the membrane is guided to a hot air oven at about 210°F (99°C) to evaporate any residual solvents.

#### 1.1 Example 1.1: FLUX and NaCl REJECTION

The membrane formed in Example 1 was tested under the following conditions:

A solution of 4000 µS (2000 ppm) NaCl in water was passed across the surface of the membrane of 1 x 3" at a pressure of 250 psi on one side of the membrane while the other side was maintained at atmospheric pressure. A temperature of 78-81°F (25-27°C) was maintained with a measured pH of 5.8-5.9. The feed rate was 2.2-2.25 gpm (8.3-8.5 lpm). The process volume filtered through (flux) was 217.20 gfd, and the percent rejection was 34.60%. The same experiment was performed at a pressure of 200 psi. The process volume filtered through was 180.76 gfd, and the percent rejection was 34.29%.

When the same experiment was performed at an adjusted pH of 7, and the pressure on one side of the membrane was 180 psi, the process volume filtered through was 173.11 gfd, and the percent rejection was 48.49%.

#### III. Prior Art Cited by the Examiner

I have reviewed U.S. Patent No. 4,983,291 to Chau et al. ("the Chau reference") and U.S. Patent No. 6,245,234 to Koo et al. ("the Koo reference"). The examples of the Chau reference show fluxes of an aqueous sodium chloride solution through a filtration membrane in the range of 4.4 to 66.7 gfd. The examples of the Koo reference show fluxes of an aqueous sodium chloride solution through a filtration membrane in the range of 17.8 to 94.7 gfd.

#### VI. Summary

There has been a longstanding need for increasing the overall fluid fluxes of filtration membranes. Surprisingly and unexpectedly, the membranes of the present invention significantly enhance the fluid flux through the membrane. For example, the

fluxes shown are greater than in the references cited by the Examiner in connection with the above-referenced application.

Thus, the methods of the invention are not obvious from the prior art.

VI. I declare further that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code and such willful false statements may jeopardize the validity of the instant patent specification or any patent issuing thereon.

By

  
John Simonetti

Date

7/19/04